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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Re application of

: **Confirmation No. 1556**

Hiroya OKUMURA et al.

: Attorney Docket No. 2001_1255A

Serial No. 09/950,081

: Group Art Unit 1745

Filed September 12, 2001

: Examiner Mark Ruthkosky

SEPARATOR FOR SOLID POLYMER TYPE
FUEL CELL AND PROCESS FOR PRODUCING
THE SAME

Mail Stop: Amendment

SUBMISSION OF EXECUTED DECLARATION

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

THE COMMISSIONER IS AUTHORIZED
TO CHARGE ANY DEFICIENCY IN THE
FEES FOR THIS PAPER TO DEPOSIT
ACCOUNT NO. 23-0975

Sir:

Submitted herewith is the original executed Declaration of Mr. Takabatake a copy of which was previously filed on September 27, 2005.

Favorable consideration and allowance is solicited.

Respectfully submitted,

Hiroya OKUMURA et al.

By: _____

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In re Application of
Hiroya OKUMURA et al.

Serial No. 09/950,081

Group Art Unit 1745

Filed September 12, 2001

Examiner Mr./Mrs. Mark RUTHKOSKY

For: SEPARATOR FOR SOLID POLYMER TYPE FUEL CELL AND
PROCESS FOR PRODUCING THE SAME

DECLARATION UNDER RULE 132

Honorable Commissioner of Patent and Trademarks,
Washington, D.C.

Sir,

I, Koji TAKABATAKE, declare:

That I am a citizen of Japan, residing at 3-6-19-
311 Nyoidani, Minoo-shi, Osaka, Japan;

That I was born on December 2, 1949 in Okayama and
graduated from the Department of Synthetic Chemistry,
Faculty of Engineering Science, Osaka University,
Toyonaka-shi, Osaka, Japan, 560 in March 1972;

That I have been employed by Nippon Shokubai
Kagaku Kogyo Co., Ltd. (current NIPPON SHOKUBAI Co.,
Ltd.), JAPAN since 1972, and engaged in the research in
thermosetting resins and sheet, and bulk molding
compounds;

That I have been on loan to Japan Composite Co.,
LTD. which is a joint venture company of Mitsui Takeda
Chemicals, Inc. and NIPPON SHOKUBAI Co., Ltd. since
April 2003, and engaged in the research in separators
for fuel cell, which was taken over from Mitsui Takeda

Chemicals, Inc., as Senior Chief Researcher since April 2004; and

That the following experiments were conducted under my direct supervision;

EXPERIMENT

I investigated effectiveness of pressurized kneading on compounds and molded articles thereof.

Sample A

In accordance with Sample 23648 recited in Examples of US 6,251,308 (Butler), the sample plate was produced. The components used were as follows:

(1) Resin-G-simulated resin

Since the "Resin G" used in Sample 23648 is manufactured by our competitive company, Rechhold Chemicals Inc., we could not obtain Resin G. Thus, we produced and used an alternate resin equivalent to Resin G.

That is, to a four-necked flask equipped with a stirrer, a condenser, a nitrogen gas inlet, and a thermometer were charged 956 g of a bisphenol-A-type epoxy resin (manufactured by Toto Kasei Co. Ltd., epototo YD-7011R, epoxy equivalent 478 g/eq), 172 g of methacrylic acid, 0.2 g of triphenylphosphine, 0.1 g of hydroquinone as a thermal polymerization inhibitor, and reacted for 8 hours at 120°C to obtain 1128 g of vinyl ester resin having an acid value of 11.6 mgKOH/g. The vinyl ester resin was diluted with 886 g of styrene monomer to obtain a Resin-G-simulated vinyl ester resin (2014 g). The Resin-G-simulated vinyl ester resin had 56 wt% solid and 520 cps of viscosity. Since the Resin-

G-simulated vinyl ester resin was quite similar to Resin G regarding solid concentration and viscosity as shown in the below Table A, we recognized that the Resin-G-simulated vinyl ester resin fell under Resin G.

Incidentally, the property of Resin G is mentioned as "Resin G is 9100 from Rechhold Chemicals, Inc., ... a bisphenol-epoxy vinyl ester ... diluted to 54-58 wt% solids with styrene" by Butler. Further, the catalog of Rechhold Chemicals, Inc. describes the resin 9100 has a viscosity of 500 cps and a solid concentration of 56.0 wt%.

Table A

	Resin G	Resin-G-simulated resin
Resin Type	Bisphenol-epoxy vinyl ester	Bisphenol-epoxy vinyl ester
Solid Concentration	56 wt%	56 wt%
Viscosity	500 cps	520 cps

(2) Rheological modifier

The "Modifier B" (polyisocyanate, "40-7263") used in the Sample 23648 produced from Cook Composites And Polymers Co. was used.

(3) Graphite

Since the "Graphite A" (having less than 10% greater than 150 μm and less than 10% smaller than 44 μm in diameter) was hardly obtainable, an alternate graphite "SPG 100", which had the mean particle size of 125 μm was used. Since the mean particle size of 125 μm is in a range of between 44 and 150 μm of Graphite A, the alternate graphite is considered to be equivalent to Graphite A.

(4) Other components

The same components in the Sample 23648 were used as follows:

Initiator: tert-butyl peroxy isopropyl carbonate

Inhibitor: 2,6-di-tertbutyl-p-cresol

Mold releasing agent: calcium stearate.

According to the formulation in Table B, the molding composition was prepared by adding the resin (Resin-G-simulated vinyl ester resin), monomer initiator (tert-butyl peroxy isopropyl carbonate), inhibitor (2,6-di-tertbutyl-p-cresol), mold release agent (calcium stearate), and rheological modifier (polyisocyanate, "40-7263") to a disperser and blending for 2 minutes.

To a kneader (manufactured by Toshin Co., Ltd., THM0.5-3 type hybrid mixer, orifice of 10 cm x 9 cm, depth of 15 cm) kneading under an atmospheric pressure, the half amount of conductive filler (Graphite "SPG 100" manufactured by SEC Co. Ltd., mean particle size of 125 μ m) was added, and with kneading constantly, the above molding composition and the resultant conductive filler were further added and mixed for 15 minutes to obtain a kneaded compound A. When mixing was complete, the kneaded compound A was put in a suitable barrier bag and allowed to mature for approximately one day before molding.

The molding parameters for the molding compositions were as follows: Molding temperature for

plaques was 295°F (146°C) with a molding time of 3 minutes under 100 kgf/cm² (approximately 9.8 MPa) to obtain a molded plate A (300 mm x 300 mm x 2 mm).

Sample B

A kneaded compound B was obtained in the similar manner to the kneaded compound A except that 117.62 g of the Resin-G-simulated vinyl ester resin without the modifier was employed instead of 100 g of the same resin and 17.62g of the modifier, and kneaded under the pressure of 4 kgf/cm² (3.92×10^5 Pa). Incidentally, the ratio of the graphite relative to the resin without the modifier in Sample B is the same with the ratio of the graphite relative to the total amount of the resin and the modifier in Sample A.

For the kneaded compounds and the molded plates obtained in Samples A and B, the following properties were evaluated.

(Kneadability)

The kneadability was visually evaluated by change of compound state at immediately after kneading, and one day after kneading.

(Electric property)

The molded plate was immediately subjected to clip between two billets each 10 mm thick at a room temperature, and allowed to stand for 30 minutes for cooling. The cooled plate was cut for obtaining a test specimen.

The test specimen was subjected to determine the electric property (volume resistivity) according to

Japanese Industrial Standards K 7194.

The results are shown in Table B.

Table B

	Samples	
	A	B
Pressurized kneading	Not conducted	conducted
Formulation		
Resin-G-simulated vinyl ester resin [g]	100	117.62
Modifier B: polyisocyanate [g]	17.62	0
Graphite [g]	378.6	378.6
Initiator: tert-butyl peroxy isopropyl carbonate [g]	2.02	2.02
Inhibitor: 2,6-di-tertbutyl-p-cresol [g]	0.5	0.5
Releasing agent: calcium stearate [g]	6.06	6.06
Properties		
Kneadability	Immediately after kneading	Coarse particle Clay-like
	One day after kneading	Thickened and hardened Clay-like
Electric property (volume resistivity) [mΩcm]	13.7	5.3

EVALUATION

As apparent from Table B, the kneaded compound A was coarse particle state immediately after kneading, and thickened and hardened one day after kneading. However, the kneaded compound B was clay like state immediately after kneading as well as one day after kneading. Therefore, it is apparent that the pressurized kneading improves the kneading efficiency of the mixture and results in good productivity.

Further, although the ratio of the graphite relative to the resin (or the resin and the modifier) was the same between Samples A and B, the volume resistivity of Sample A was over 2.5 times higher than that of Sample B.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Signed this 14th day of February, 2006

A handwritten signature in cursive script, reading "Koji Takabatake". The signature is written in dark ink and is positioned above the printed name.

Koji TAKABATAKE